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AUTHORITY

USNWC ltr, 30 Aug 1974

IDEP FORM 12 11 82 IDEP Q4243

SPECIFICATION

D0727

1. COMPONENT/PART NAME PER GENERIC CODE	2. PROGRAM OR WEAPON SYSTEM	3. DATE OF:	DAY	MO	YR
Propulsion Parts & Materials, Solid Fuel Engines, Propellants	Multiple	ISSUE	13	10	67
4. ORIGINATOR'S SPECIFICATION TITLE: Purchase Description - Toluene -2, 4-Diamine	5. ORIGINATOR'S SPEC. NO.: WS 7650	REVISION			

6. SPECIFICATION IS: ☐ DRAFT ☐ PRELIMINARY ☒ FINAL

7. THIS SPECIFICATION COMPLEMENTS REPORT NO:

8. TYPE OF SPECIFICATION

- ☒ (A) GENERAL PRODUCT REQUIREMENTS FOR A FAMILY OF PARTS - PROCUREMENT DOCUMENT
- ☐ (B) INDIVIDUAL DETAIL PARTS DOCUMENT; STDS BOOK PAGES - FOR PROCUREMENT
- ☐ (C) DETAIL INSPECTION, PROCESS CONTROL, AND/OR TEST PROCEDURES FOR SPECIFIC PARTS
- ☐ (D) PROCESS (PAINTING, WELDING, FINISHING, HEAT TREATING ETC.) APPLICABLE TO MANY PARTS
- ☐ (E) SPEC FOR PERFORMANCE, RELIABILITY, AND/OR ENVIRONMENT FOR ASSEMBLIES, EQUIPMENTS, SUBSYSTEMS AND SYSTEMS
- ☐ (F) PERFORMANCE AND APPLICATION DATA FOR DESIGN ENG USE ON PARTS - NOT FOR PROCUREMENT
- ☐ (G) OTHER (DETAIL IN 10.)

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9. APPLICABLE REFERENCED SPECIFICATIONS EXCEPT MIL-SPECS, STDS, OR OTHER UNIVERSALLY AVAILABLE DOCUMENTS.	ENCL	AVAIL ON REQ	NOT AVAIL	PREVIOUSLY SUBMITTED AND FILED AS IDEP REPORT OR SPEC NUMBER
1961 Book of ASTM Std, Part 15 & 27			x	

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13. SPEC. NO 565,70,00,00-X7-1

11. SIGNED	12. CONTRACTOR	SUBCONTRACTOR
R. S. Harper 11/18/68	NWC/CL	

17 61 11

NOTICES PAGE

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Code Ident
30003

WS 7656

NAVAL AIR SYSTEMS COMMAND

DEPARTMENT OF THE NAVY

PURCHASE DESCRIPTION

TOLUENE -2,4-DIAMINE

1. SCOPE.

1.1 Scope. This purchase description covers a technical grade of toluene -2,4-diamine.

2. APPLICABLE DOCUMENTS.

2.1 The following document of the issue in effect on date of invitation for bids or request for proposal forms a part of this document to the extent specified herein.

STANDARDS

Military

MIL-STD-129 Marking for Shipment and Storage.

(Copies of specifications, standards, drawings, and publications required by suppliers in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

FSC 6810

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2.2 Other publications. The following documents form a part of this document to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposal shall apply.

American Society for Testing and Materials (ASTM)

1961 Book of ASTM Standards; Part 27	Test-Method ASTM D1303-55 "Total Chlorine in Vinyl Chloride Polymers and Copolymers".
1963 Book of ASTM Standards; Part 15	Test-Method ASTM D871-63, "Testing Cellulose Acetate".

(ASTM Publications are published by the American Society for Testing and Materials, Philadelphia 3, Pennsylvania.)

3. REQUIREMENTS.

3.1 Preproduction sample. Unless otherwise specified (see 6.2), a preproduction sample shall meet all requirements of this document. The preproduction sample shall be prepared using the same methods and procedures proposed for production. Any production prior to acceptance of the preproduction sample shall be at the risk of the supplier.

3.2 Data. No data is required by this document or by referenced documents in section 2 unless specified in the contract or purchase order.

3.3 Compliance to documents. Toluene -2,4-diamine shall conform to the requirements herein and to the applicable requirements of documents listed in section 2.

3.4 Product characteristics and performance. When tested in accordance with 4.7 of this document, toluene -2,4-diamine shall meet the following product characteristics and performance.

3.4.1 Chemical and physical analysis. The chemical and physical analysis of the material shall be as specified in Table I.

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Table I. Chemical and Physical Analysis

Characteristics	Minimum	Maximum
Toluene -2,4-diamine, %	99.0	----
Water insolubles, %	----	0.10
Chlorine, %	----	0.10
Melting point, °C	97.0	----
Moisture, %	----	1.0
Ash, %	----	0.20
Acid insolubles, %	----	5.0

3.5 Workmanship. The toluene -2,4-diamine shall be uniform in quality, free from foreign materials, and shall be manufactured under conditions and procedures standard in the industry.

4. QUALITY ASSURANCE PROVISIONS.

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own facilities or any commercial laboratory acceptable to the Government. The Government reserves the right to perform any of the inspections set forth in this document where such inspections are deemed necessary to assure that supplies and services conform to prescribed requirements.

4.2 Lot. A lot shall consist of material produced at one plant with no change in formulation or process. If manufacture is by batch process, each batch shall constitute a lot. A batch shall be as defined in 6.3.

4.3 Acceptance sampling. The number of containers to be chosen at random for acceptance sampling shall be equal to the square root of the total number of containers in the lot. If the number thus obtained is not a whole number, the number of containers to be sampled shall be increased

to the next higher whole number. In no case, however, shall the number of containers to be sampled be less than seven (unless there are less than seven containers in the lot, in which case, each container shall be sampled).

4.3.1 Primary sample. From each selected container, a sample shall be taken from three or more places throughout the container. The total weight of the samples taken from each container shall weigh at least 50 grams (gm). Each sample thus taken shall be mixed thoroughly, placed in a clean dry container, and labeled to identify the material name, original container designation, contract number, and lot number.

4.3.2 Composite sample. Each primary sample shall be subdivided to prepare a composite sample (not in excess of 500 gm). Primary material not used shall be returned to the primary sample container. After mixing the composite sample thoroughly, the composite sample shall be placed in a clean, dry container and sealed. The composite sample shall be identified with the material name, container designation, contract number, and lot number. All specified chemical tests shall be made on this composite sample representing the lot. Failure of the composite sample to pass all of the tests herein shall result in rejection of the lot represented.

4.4 Classification of tests. Inspection and testing of toluene -2,4-diamine shall be classified as follows:

(a) Preproduction tests.

(b) Quality conformance tests.

4.5 Preproduction tests. Preproduction tests shall be conducted only on the preproduction sample and shall consist of all examinations and tests specified in 4.6.

4.6 Quality conformance tests. Quality conformance tests for acceptance of the toluene -2,4-diamine shall consist of the following tests:

Characteristics	Tests
Toluene -2,4-diamine	4.7.1
Water insolubles	4.7.2
Chlorine	4.7.3
Melting point	4.7.4
Moisture	4.7.5
Ash	4.7.6
Acid insolubles	4.7.7

4.7 Tests. The following procedures shall be used to determine that the requirements of this document have been met. Any proposed change in test procedures or equipment shall necessitate, before adoption, prior approval of the procuring activity. In case of dispute between the results from any proposed method or equipment and what is cited herein, the results using the methods and the equipment specified in this document shall prevail. Unless otherwise specified, all tests shall be run in duplicate. The average of the two results shall be taken as the test result.

4.7.1 Toluene -2,4-diamine.

4.7.1.1 Principle. The method for the determination of the purity of toluene -2,4-diamine depends upon a titration with diazo p-chlorobenzene solution in an acetic acid medium. This method shall be used for analysis of technical grade toluene -2,4-diamine.

4.7.1.2 Diazotized p-chloroaniline (diazo p-chlorobenzene) Standard solution.

(1) Reagents.

(a) Para-chloroaniline, technical grade

(b) Concentrated hydrochloric acid

(c) Sulfamic acid, technical grade

(d) Potassium bromide crystals, analytical grade

(e) Potassium bromate-potassium bromide ($KBrO_3$-KBr) solution 1/3 Normal (N/8), standardized

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(2) Procedures.

(a) Preparation of 0.5 N p-chloroaniline hydrochloride solution. To prepare one liter of 0.5 N solution, dissolve approximately 63.8 gm of p-chloroaniline in 500 milliliter (ml) of distilled water containing 100 ml of concentrated hydrochloric acid. Heat, if necessary, to complete the dissolution. Add a small quantity of activated charcoal, mix well and filter. Pour the filtrate into a one liter volumetric flask and dilute to volume. (After standardization, store in an amber colored flask.)

(b) Standardization of the 0.5 N p-chloroaniline hydrochloride solution. Pipet 50 ml of the p-chloroaniline solution into a one liter volumetric flask and dilute to volume. Mix thoroughly and pipet a 50 ml aliquot into a 400 ml beaker. Add 200 ml of distilled water and 15 ml of hydrochloric acid. Titrate with N/8 $KBrO_3$-KBr solution adding the $KBrO_3$-KBr solution drop-wise until a spot test with potassium (or cadmium) iodide-starch papers shows a faint distinct spot for 2 minutes. Make a blank determination omitting only the p-chloroaniline.

$$\text{Normality of p-chloroaniline} = \frac{(A-B) \times N}{10}$$

Where: A = Volume of $KBrO_3$-KBr used to titrate sample, ml
B = Volume of $KBrO_3$-KBr used to titrate blank, ml
N = Normality of $KBrO_3$-KBr

(c) Preparation of N/40 solution of diazo p-chlorobenzene hydrochloride solution. Pipet 50 ml of 0.5 N p-chloroaniline solution into a 600 ml beaker. Add 300 ml of distilled water, 10 ml of concentrated hydrochloric acid and about 10 gm of potassium bromide. Then add, by rapid pouring, the measured amount of 0.5N sodium nitrite solution

required to diazotize the p-chloroaniline plus 1.0 ml in excess. Stir thoroughly, let stand 10 to 15 minutes, then add saturated sulfamic acid solution drop-wise until no excess sodium nitrite is indicated when spotted on potassium (or cadmium) iodide-starch indicator papers. Transfer the diazo solution into an amber colored 1000 ml volumetric flask and dilute to volume.

$$\text{Normality of the diazo p-chlorobenzene solution} = \frac{\text{Normality of p-chloroaniline hydrochloride solution}}{20}$$

4.7.1.3 Procedure. Weigh out a sample equivalent to 0.25 to 0.27 gm of 100 percent toluene -2,4-diamine, transfer to a 600 ml beaker and dissolve in about 75 ml of distilled water. Add 5 ml of glacial acetic acid, 20 gm of sodium acetate free from carbonates, and 20 gm of sodium chloride. Dilute to the 300 ml mark with distilled water and titrate at room temperature, about 25 degrees centigrade (°C) (77 degrees Fahrenheit (°F)), with N/40 diazo p-chlorobenzene solution. Follow the titration for the disappearance of toluene -2,4-diamine by spot testing against the diazo p-chlorobenzene solution and for excess of diazo solution by spot testing (see 4.7.1.4) against a saturated solution of toluene -2,4-diamine. The reaction is complete when no line is formed with the diazo spot and a positive end point against toluene -2,4-diamine persists for 3 minutes. The diazo p-chlorobenzene solution should be added in 0.1 ml portions near the end point. During the titration, lines may be obtained with both indicators. This indicates that the temperature of the sample is too low or the diazo solution is being added too rapidly. In such cases, make the necessary adjustments and do not add any diazo solution until one of the spot tests has disappeared.

$$\text{Percent toluene -2,4-diamine} = \frac{A \times B \times 12.22}{C}$$

Where: A = Volume of diazo solution used in titration, ml
B = Normality of diazo solution
C = Weight of sample, gm

4.7.1.4 Notes. Follow the reaction by means of what is called a "negative" spot test. Make a line about 1 inch long upon a strip of spot test paper (Reeve Angel Filter Paper) with a stirring rod moistened with the titrated solution and allow it to stand about 15 seconds, or until a colorless rim has formed around the line. Then moisten the end of another stirring rod with the diazo solution and make a parallel line on the spot test paper about 1/2 inch from the first line. The rim of this line should spread into the first line. As long as the uncoupled sample remains in solution, color will be produced at the point of contact of the two lines. When the coupling is practically complete, no color will be formed and testing for the end point is commenced. The end point or "positive" spot test is detected by means of a saturated solution of toluene -2,4-diamine in distilled water. Make a line about 1 inch long upon a spot test paper with a stirring rod moistened with the toluene -2,4-diamine solution. Make a similar and parallel line with the solution being titrated about 1/2 inch from the indicator line. The colorless rim of the dye spot will then spread into the toluene -2,4-diamine and a trace of color formed at the junction indicating the completion of the coupling and the presence of a slight excess of diazo solution.

4.7.1.5 Acceptance criteria. For the lot represented to pass the toluene -2,4-diamine assay test, the value obtained for the percent toluene -2,4-diamine shall be not less than the value specified in 3.4.1.

4.7.2 Water insolubles.

4.7.2.1 Procedure. Dissolve approximately 20 gm of sample (weighed to the nearest 0.001 gm) in one liter of distilled water. Filter the solution by suction through a fritted glass funnel.

$$\text{Percent water insoluble} = \frac{B \times 100}{A}$$

Where: A = Weight of sample dissolved in distilled water, gm
B = Weight of residue collected on filter, gm

4.7.2.2 Acceptance criteria. For the lot represented to pass the water insoluble test, the value obtained for the percent water insoluble shall be no greater than the value specified in 3.4.1.

4.7.3 Chlorine. The percentage of chlorine specified in 3.4.1 shall be determined in accordance with the procedure given in Test Method D1303-55,"Total Chlorine in Vinyl Chloride Polymers and Copolymers" (Part 27 of ASTM, page 472), except use ferric alum indicator in place of a ferric nitrate indicator, and the electrical ignition of the Parr bomb in place of ignition by use of a Bunsen burner.

4.7.3.1 Acceptance criteria. For the lot represented to pass the chlorine test, the value obtained for the percent chlorine shall be no greater than the value specified in 3.4.1.

4.7.4 Melting point.

4.7.4.1 Apparatus. Melting Point Apparatus, Fisher-Johns, available from the Fisher Scientific Company, Pittsburg, Pennsylvania.

4.7.4.2 Procedure. Place a few crystals of toluene -2,4-diamine on an 18 millimeter diameter micro-cover glass. Cover the crystals with an identical cover glass and transfer to the cleaned heating stage of a Fisher-Johns melting point apparatus (4.7.4.1). Turn on the apparatus, bring the temperature to 90°C (194°F) and hold the apparatus at that temperature for at least five minutes. Then adjust the reading of the current-input dial to give a temperature rise rate of 0.8 to 1.0°C (1.4 to 1.8°F) per minute as indicated by the attached thermometer. With the aid of the illuminating and magnifying unit, observe the melting point in degrees centigrade at which the last crystal of toluene -2, 4-diamine liquifies.

4.7.4.3 Acceptance criteria. For the lot represented to pass the melting point test, the value obtained for the melting point in degrees centigrade shall be not less than the value specified in 3.4.1.

4.7.5 Moisture.

4.7.5.1 Apparatus. The apparatus used for determination of moisture content of the sample shall be an Aquameter, Model KF-2 or KF-3, Beckman Instruments, Inc., Fullerton, California, or an approved equivalent. The Aquameter shall be prepared for operation as described in the technical manual furnished by the manufacturer (Beckman Instruments, Inc.). Use of an alternate equivalent item or equipment approved by the procuring activity will necessitate use of the specific technical manual prepared by the manufacturer.

4.7.5.2 Reagents.

(a) Karl Fischer reagent. Karl Fischer reagent must have a strength such that each milliliter of Karl Fischer reagent corresponds to 0.0014 to 0.0023 gm of distilled water. Dilute 750 ml of commercially available stabilized Karl Fischer reagent (with water equivalent of 0.005 to 0.007 gm/ml) to 2000 ml with absolute methanol (0.1 percent water, maximum). Mix well and allow to stand overnight before use. Determine the water equivalent (A) of this solution as follows:

1. Use sodium tartrate dihydrate ($Na_2C_4H_4O_6 \cdot 2H_2O$) as a primary standard (with a water content of 15.66 percent) for standardizing Karl Fischer reagent. If the water content value is in question, it may be determined by heating some of the salt at 150°C (302°F) for three hours. Should the value (as determined) differ from the theoretical value of 15.66, then the experimental value shall be used in determination of water equivalent (A) of the Karl Fischer reagent; i.e., instead of the 15.66 in the formula below, the factor should be 10P where P is percentage moisture (as determined). Rapidly transfer 0.090 to 0.110 gm (weighed to the nearest 0.0001 gm) of reagent-grade $Na_2C_4H_4O_6 \cdot 2H_2O$ to the titration vessel.

2. Titrate to an end point in the same manner as with the sample (see 4.7.5.3).

3. Repeat the standardization procedure until three successive results agree within five parts per thousand.

4. If the indicated water equivalent (A) of the Karl Fischer reagent is less than 0.0014 gm of water per ml of Karl Fischer reagent, it may be due to the presence of too much water in the absolute methanol used. In this case, distill the methanol from metallic calcium or calcium hydride. Passing the methanol through a column of Molecular Sieves, Type 4A, may also reduce the water content of the methanol sufficiently. (Molecular Sieves are a product of the Linde Company, a division of Union Carbide Corporation, New York City, New York.)

$$\text{Water equivalent (A)} = \frac{0.1566\ W}{V}$$

Where: A = Water equivalent of the Karl Fischer reagent, gm/ml
W = Weight of $Na_2C_4H_4O_6 \cdot 2H_2O$ taken, gm
V = Volume of Karl Fischer reagent used, ml

(b) Water-in-methanol solution. The water-methanol solution should contain 0.0015 to 0.0020 gm of water per ml of solution. A good grade of commercial absolute methanol contains about 0.0010 gm water per ml of methanol. Water content can be adjusted by adding 1.0 gm of water to 1000 ml of the water-methanol solution in terms of Karl Fischer reagent as follows:

1. Put about 50 ml of the anhydrous methanol used in 4.7.5.2 (a) into the titration beaker of the Aquameter. Add a slight excess of Karl Fischer reagent (4.7.5.2 (a)), then back titrate with water-methanol solution

(4.7.5.2 (b)). Then run in an additional 5 to 8 ml of Karl Fischer reagent, read to the nearest 0.01 ml, and again back titrate with water-methanol solution (read to the nearest 0.01 ml). Repeat the addition and back titrating steps twice more to provide triplicate determinations of the equivalency ratio. Calculate the ratio (B) of Karl Fischer reagent to that of the water-methanol solution. The range of the ratios calculated from the three titrations should not be greater than 0.04. If the range exceeds 0.04, continue making titrations until three ratios are obtained whose range does not exceed 0.04. Then determine the average ratio from all the ratios which have been obtained.

4.7.5.3 Procedure. Determine the moisture content of the sample by the Karl Fischer method using a direct-titration technique. Introduce to the titration beaker, through the opening in the diaphragm, approximately 10 gm of sample weighed to the nearest 0.001 gm and a mixture of 150 ml of anhydrous methanol with 50 ml of glacial acetic acid. Both materials are to be reagent grade. Close the opening, start the stirrer and press the titration button to titrate with Karl Fischer reagent (4.7.5.2 (a)). When the indicator light glows, read the Karl Fischer buret to the nearest 0.01 ml. Where necessary water-methanol solution (4.7.5.2 (b)) may be used to back titrate.

$$\text{Percent moisture} = \frac{100A\ (V_{KF} - BV_{WM})}{W}$$

Where:

A = Weight of distilled water equivalent to 1.00 ml of Karl Fischer reagent, gm/ml

V_{KF} = Volume of Karl Fischer reagent titrant used, ml

V_{WM} = Volume of water-methanol solution titrant used, ml

B = Ratio of Karl Fischer reagent to that of water-methanol solution, ml/ml

W = Weight of sample taken, gm

4.7.5.4 Acceptance criteria. For the lot represented to pass the moisture test, the value obtained for percent moisture shall be no greater than the value specified in 3.4.1.

4.7.6 Ash. The percentage of ash specified in 3.4.1 shall be determined in accordance with the procedure given in Test Method D871-63,"Testing Cellulose Acetate" (Part 15 of ASTM, page 271).

4.7.6.1 Acceptance criteria. For the lot represented to pass the ash test, the value obtained for percent ash shall be no greater than the value specified in 3.4.1.

4.7.7 Acid insolubles.

4.7.7.1 Procedure. Dissolve approximately 1 gm of sample (weighed to the nearest 0.001 gm) in 100 gm of 5 percent hydrochloric acid at room temperature. Filter the solution by suction through a fritted glass funnel.

$$\text{Percent acid insoluble} = \frac{B \times 100}{A}$$

Where: A = Weight of sample dissolved in hydrochloric acid, gm
B = Weight of residue collected on filter, gm

4.7.7.2 Acceptance criteria. For the lot represented to pass the acid insoluble test, the value obtained for percent acid insoluble shall be no greater than the value specified in 3.4.1.

4.8 Packing and marking. Determine that all packing and marking conforms to section 5 of this document.

5. PREPARATION FOR DELIVERY.

5.1 Preservation and packaging. Not applicable (unless specified in the contract or purchase order).

5.2 Packing.

5.2.1 Level A. Not applicable.

5.2.2 Level B. Not applicable.

5.2.3 Level C. The material shall be packed as directed in the contract to afford protection against damage during direct shipment from the supply source to the first receiving activity for immediate use. Containers shall comply with common carrier regulations applicable to the mode of transportation to be used. (See 6.2.)

5.3 Marking. In addition to the markings required by contract or order, unit packages and shipping containers shall be marked in accordance with the requirements of MIL-STD-129.

6. NOTES.

6.1 Intended use. Toluene -2,4-diamine described in this document is intended for use as a stabilizer in ammonium-nitrate-based solid propellants.

6.2 Ordering data. Procurement documents should specify the following:

(a) Title, number and date of this document.

(b) Whether a preproduction sample is required (see 3.1).

(c) Type and size of shipping container (see 5.2.3).

6.3 Definition.

6.3.1 Batch. A batch is defined as that quantity of material which has been subjected to one or more chemical or physical processes (or combinations thereof) intended to produce a desired product having substantially uniform characteristics. The final step in the processing must have treated the entire contents of the batch at one time.

6.4 Safety and health warning. Handling of the chemicals specified herein shall be in accordance with suitable safety and health precautions.

6.5 Approved product. An approved product under this document is toluene-2,4 diamine, technical grade, manufactured by E.I. Dupont de Nemours Co., Inc., Chicago, Illinois.

Custodian:
NASC 52021E

Preparing Activity:
NWC/China Lake, California